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| 1. REPORT DATE *(DD-MM-YYYY)* | 2. REPORT TYPE | 3. DATES COVERED *(From – To)* |
|---|---|---|
| 19-02-2002 | Final Report | 29 October 2001 - 29-Oct-02 |

**4. TITLE AND SUBTITLE**

~~Non-Destructive Evaluation Using SQUID~~

**5a. CONTRACT NUMBER** F61775-02-WD078

**5b. GRANT NUMBER**

**5c. PROGRAM ELEMENT NUMBER**

**6. AUTHOR(S)**

Associate Professor Hamid Reza Kokabi

**5d. PROJECT NUMBER**

**5d. TASK NUMBER**

**5e. WORK UNIT NUMBER**

**7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)**

Université Pierre et Marie Curie (Paris 6)
Tour 12, 2e étage
Paris 75252
France

**8. PERFORMING ORGANIZATION REPORT NUMBER**

N/A

**9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)**

EOARD
PSC 802 BOX 14
FPO 09499-0014

**10. SPONSOR/MONITOR'S ACRONYM(S)**

**11. SPONSOR/MONITOR'S REPORT NUMBER(S)**

SPC 01-4078

**12. DISTRIBUTION/AVAILABILITY STATEMENT**

Approved for public release; distribution is unlimited.

20040625 089

**13. SUPPLEMENTARY NOTES**

**14. ABSTRACT**

This report results from a contract tasking Université Pierre et Marie Curie (Paris 6) as follows: The contractor will investigate the use of high sensitivity, nondestructive, SQUID magnetometry to detect and understand crack formation and propagation in composites-based organic matrix materials, as well as in aluminum and carbon-composite material used in aircraft structures.

**15. SUBJECT TERMS**

EOARD, Radio Propagation, Superconductivity, Non-destructive inspection

| 16. SECURITY CLASSIFICATION OF: a. REPORT | b. ABSTRACT | c. THIS PAGE | 17. LIMITATION OF ABSTRACT | 18, NUMBER OF PAGES | 19a. NAME OF RESPONSIBLE PERSON |
|---|---|---|---|---|---|
| UNCLAS | UNCLAS | UNCLAS | UL | 2 | David M. Burns, Lt Col, USAF |
| | | | | | **19b. TELEPHONE NUMBER** *(Include area code)* +44 (0)20 7514 4955 |

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**DATE:** September 2, 2003

**Name and Title of Authorized Official:** KOKABI, Associate Professor

UNIVERSITE PIERRE & MARIE CURIE
LA SCIENCE A PARIS

LISIF

Laboratoire des Instruments et Systèmes de l'Ile-de-France

# *Nondestructive evaluation (NDE) of crack formation in advanced composite materials and aluminum during dynamic fatigue tests by SQUID magnetometry*

Final report of SPC 01-4078:

Contract Order Number : F61775-02-WD078

European Office of Aerospace Research and Development (EOARD)

By the principal investigator :

***Hamid KOKABI, Ph.D.***

***Associate Professor***

June 2003

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# Summary

# I. Introduction

Metallic materials and non-metallic advanced composites are widely used in different aircraft structures and components. High performance NDE methods will be of great utility for these types of materials. Because of its high sensitivity [1], SQUID magnetometry is a promising NDE method to detect flaw formation and propagation in such diamagnetic and paramagnetic materials (aluminum, composite-based organic matrix and carbon-composite materials). In fact, there have been few reports on SQUID NDE in such non-magnetic materials [2, 3, 4]. The advantages of a SQUID for NDE include high sensitivity (10-100 fT $Hz^{-1/2}$), wide bandwidth (from dc to 10 kHz), broad dynamic range (> 80dB) and its intrinsically quantitative nature.

We received our HTS rf four-SQUID Gradiometer system purchased with funds of EOARD from the Jülicher SQUID GmbH (JSQ) company at the end of June 2002. The SQUID NDE instrumentation was then set up at our laboratory with the help and advice of Professor Harold Weinstock during his one month visiting Professorship (September 9 to October 11) at our university. Then the scanning stage control card and the signal acquisition cards, purchased by our laboratory, were integrated in a PC, and LABVIEW programs have been developed to achieve an automatic NDE acquisition system.

In this report, the description of the SQUID NDE instrumentation is given in the section II. Then in section III, the noise measurements for different frequency ranges in and out of a shielded environment are presented. In section IV, we present the preliminary results obtained with specimens of aluminum obtained from the US Air Force and various composites-based organic matrix materials (filled polymers). The conclusions and the perspectives of this study will be given in section V.

# II. SQUID NDE instrumentation

The HTS rf Four-SQUID Gradiometer system that had been purchased under the present EOARD contract has the following technical specifications :

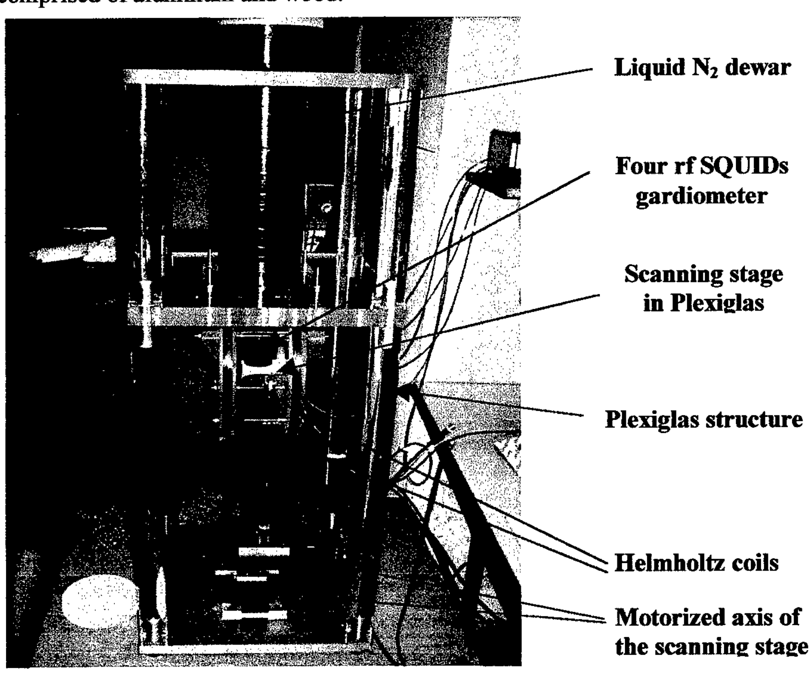
- 4 JSQ Magnetometer SQUIDs, sensitivity: 6.9 nT / $\Phi_o$, white noise: 70 $\mu\Phi_o /(Hz)^{1/2}$, Washer dia. 3.5 mm, loop 150 x 150 $\mu m^2$, operating frequency approx. 800 MHz, individually staggered for each SQUID.
- 4 JSQ SQUID readout electronics units, integrated with a Tiger microcontroller in one block
- Touchpanel control
- Power supplies for electronics block and touchpanel
- Differential electronics for manual adjustment of gradiometer signal with 3 potentiometers
- Liquid nitrogen bath cryostat
- Total lift-off of about 9 mm

The system provides a ± 10 V signal as output signal proportional to the measured field gradient, and we have connected it to a data acquisition card integrated in a PC.

## II.1. SQUID set-up

In order to avoid all magnetic perturbations from magnetic elements, the structure of the set up has been made mainly from Plexiglas, with a base comprised of aluminum and wood.

Figure 1: High Tc SQUID NDE instrumentation set - up.



Other important elements of the SQUID NDE instrumentation are:

- A scanning stage that moves along the X and Y axis with the help of two motors.
- Two Helmholtz coils for magnetic excitation used to do measurements on non-magnetic materials.
- Computer with the data acquisition card and the motors control card.
- A function generator.
- A Lock-in Amplifier.

## II.2. Scanning stage control card

The scanning stage control card is provided by STANDA company together with 3 identical motorized axes each having a total scanning rang of 100 mm with a resolution of 2.5 µm. The main characteristics of the control card are: operation of **1, 2** or **3** motors simultaneously, real-time external synchronisation signals input and output on two wires, software for Windows with functions for LABVIEW.

The three separate axes are mounted in our laboratory in a X-Y-Z reference frame. As the Z-axis motor, containing magnetic elements, created important low frequency magnetic noise during its motion, it was replaced by a manually adjustable non-magnetic axis.

The two motors are connected to the PC using the card. The movement of the motors is controlled with the LABVIEW software (see section II.5) and allows us to move the motors individually with the number of steps desired. To make a complete analysis of a sample of a particular material, we have to move the scanning stage and take measurements across the entire surface of the sample. For accurate measurements of small defects, the step between two measurement points should be very small, and thus the measurement process can be very long.

After adjusting the Z position of the sample, as near as possible to the SQUID system, the motors can move along the surface of the sample from the initial point (0,0) until the final point.

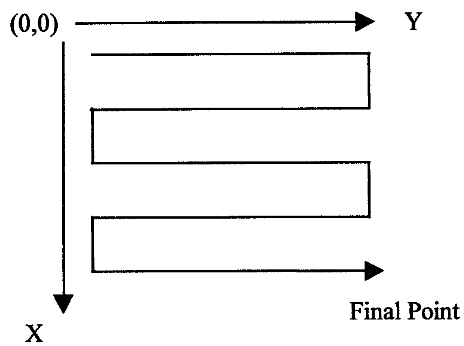



Figure 2: The X-Y movement sequence of the motors controlled by the LABVIEW program.

## II.3. Data acquisition card

The data acquisition card is provided by Keithley company with the following main characteristics :

- Resolution of the input and output signal: 16 bits.
- 16 single-ended or 8 differential channels.
- Maximum sample rate up to 100k Samples/s.
- 2 waveform-quality analog outputs.
- Maximum current: 1A.
- Maximum voltage: 30 V rms. 60VDC.

The data acquisition card is also controlled by the LABVIEW program via its special drivers. Besides it had to be calibrated with a very accurate voltage between 9 and 9.5 V using a precision voltmeter and a stabilized source because the calibration software provided for 6 decimal places. The operator can chose the range of voltage (DC or AC) measurements so that the resolution can be adjusted.

## II.4. The lock-in amplifier

After some calibration of the SQUID and measurements on magnetic samples with no magnetic excitation signals, we installed a Helmholtz coil system to do measurements with diamagnetic and paramagnetic materials. Although the SQUID system is based on the gradiometer method, the DC and low frequency noise level are quite high in the environment of our SQUID. So we applied an AC excitation signal, and a lock-in amplifier has been used to achieve a synchronous detection. The reference or excitation signal is the sine signal used to excite the Helmholtz coils in order to obtain measurement signals from the SQUID while scanning the surface of the sample. The graph in figure 3 shows the main characteristics of the lock-in amplifier.

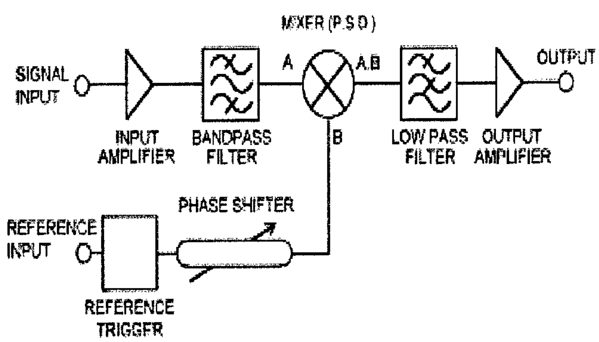



Figure 3 : The lock-in amplifier schematic diagram.

## II.5. LABVIEW programs

The entire program that will control the movement of the motors and the data acquisition has been developed with the graphical programming language LABVIEW. All the sub-programs have been optimized to automate the entire measuring process through the two axes. At the end of the measurement process we can observe the data obtained on a 3D graph on the screen. We also get a file with all the acquired data that allows us to treat the data more thoroughly.

Figure 4 shows the LABVIEW front panel program for the scanning stage control (motors X and Y) and DATA acquisition. In the "Data File" window, different information concerning the sample and the experimental conditions can be recorded at the top of the final result file. Then with the beginning of the acquisition, the X and Y positions and the mean value of the voltage measurements and its deviation are stored for each scanning point.

In the "Movement of the motors" window, we can control them first manually to bring the starting point to a reference position on the sample. As the absolute position of the motors can not be known, the relative positions of the motors are always updated and controlled with two indicators in the main window. The reset button lets one put the position indicators to 0. Then with the automatic mode and the corresponding step period a matrix is drawn with the motors. Once a motor has finished moving along the Y axis, the other motor moves one step along the X axis. The measurements are always taken when the motors are stopped.

In fact, the "Number of acquisitions at each point" allows one to average the signal for a better accuracy. We use a loop that takes several measurements from the same point. That number of measurements has been introduced by the user before starting the movement. Then, the program calculates the mean value and its deviation. Besides, it is possible to view the instantaneous mean value as well as its deviation, and a graph where we can see the real time evolution of the measurements along the X or Y axis.

Due to the fact that we are covering the surface of the sample in an X-Y meander path, for constructing the data matrix we have to rotate the order of the alternate rows. At the end of the acquisition process, we get a 3D representation of the voltage values obtained over the entire surface of the sample.

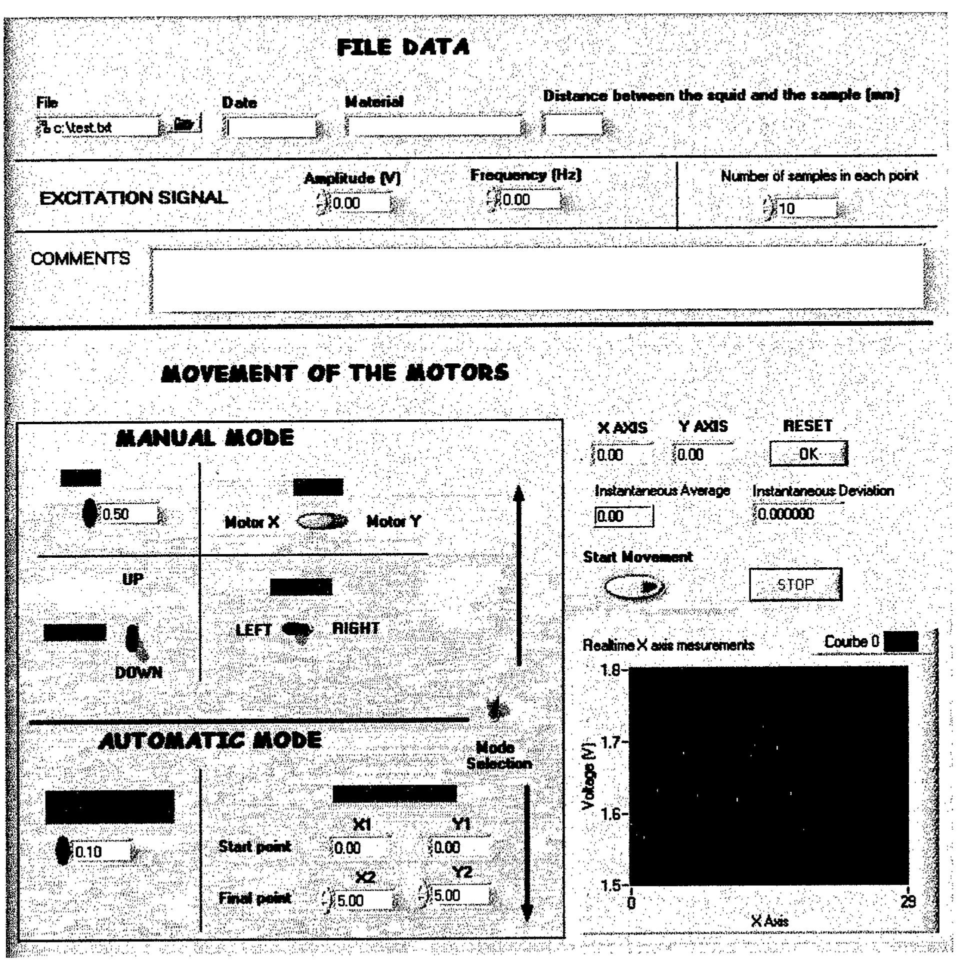



Figure 4 : LABVIEW front panel program for the scanning stage control (motors X and Y) and DATA acquisition.

## III. SQUID noise figures

With the help of Dr. Joachim Krause from JSQ company during his visit in April 2003 to replace the damaged Z2 SQUID, we measured the noise figures in and out of a shielded 3 layer mu-metal cylinder (borrowed from JSQ) of all four SQUIDs (X, Y, Z1 and Z2), and also the gradiometer for two different frequency ranges : 0-400 and 0-3200 Hz (Figure 5).

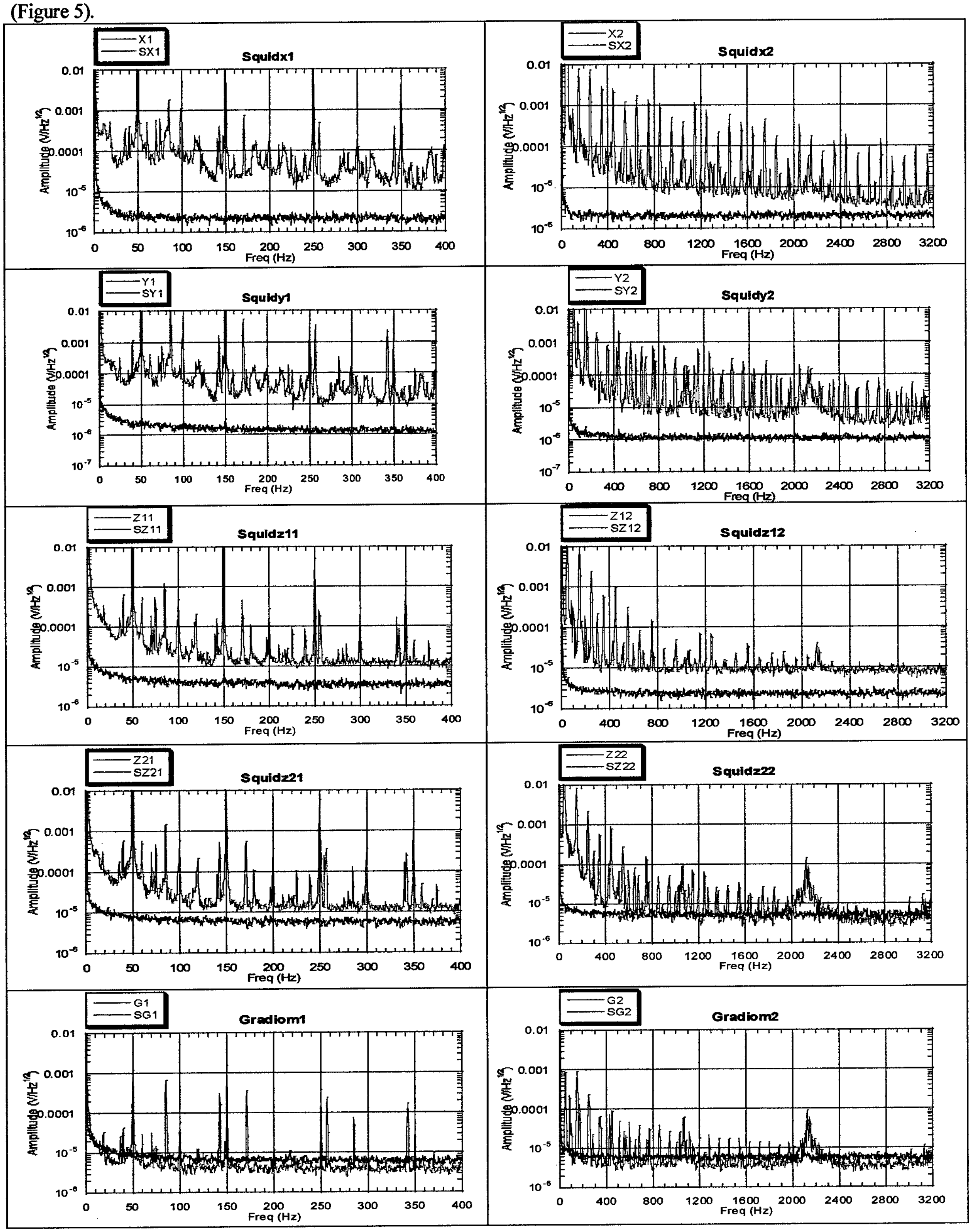



Figure 5 : Noise figures of the SQUIDs X, Y, Z1 and Z2 in and out of a shielded 3 layer mu-metal cylinder for two frequency ranges, 0-400 (left) and 0-3200 Hz (right).

# IV. Preliminary SQUID NDE results

## IV.1. Specifications of the samples

Three types of samples of different materials prepared in the shape shown in figure 6 have been examined by the SQUID in the DC and AC mode. This shape has been chosen as it is adapted for fatigue testing.

L1 = 120 mm
L2 = 175 mm
L3 = 210 mm
b1 = 10 mm
b2 = 20 mm
R = 60 mm
Thickness = 1 mm

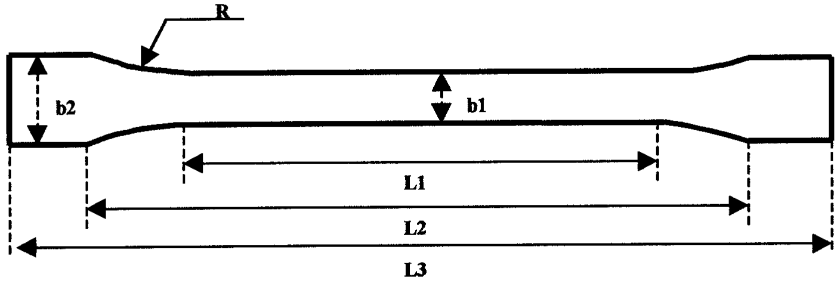



Figure 6 : The shape of the samples prepared for the SQUID NDE tests.

Aluminum samples have been provided by the US Air Force in the form of sheets cut from a C130 airplane and then transformed in the shape of the test samples (figure 6) at our laboratory. The composites-based organic matrix materials based on thermoplastics (Polystyrene as an amorphous polymer and Polypropylene as a semi-crystalline polymer) filled by the reinforcing agent of aluminum particles (30%) and carbon nanoparticles (5%) have been prepared by the project partner, Dr. Abbas Tcharkhtchi at LVTP laboratory from ENSAM institute.

Besides, thick aluminum sheets of 110 X 100 X 5 $mm^3$ dimensions with calibrated cracks in the middle of the sheet of 1mm depth and different widths (0.5, 1, 2 mm) have been fabricated and tested by our SQUID system.

## IV.2. DC measurements

We first scanned the different samples positioned very near the bottom of the SQUID system (lift-off of 0.5 mm) with no DC excitation in the Helmholtz coils and then with increasing DC magnetic field excitation up to 10 V. This voltage value corresponds to a DC magnetic field amplitude of 2.28 G. The scanning distance is 50mm with the 25mm corresponding to the exact center of the SQUID system.

The different figures (7, 8 and 9) respectively for Al sample, Al particles filled polymer and Carbon nanoparticles filled polymer show that because of the DC and low frequency noise level in the environment of our SQUID, the DC measurements can not give significant results. We would like to repeat these experiments in a shielded 3-layer mu-metal cylinder.

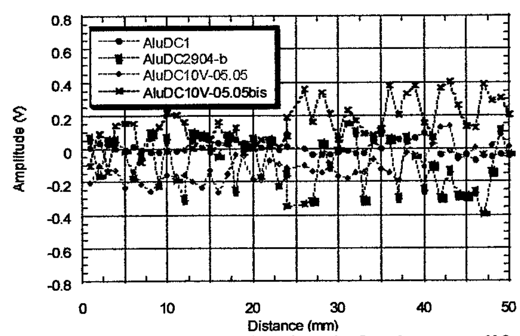



Figure 7 : Comparison of the signals for the same lift-off distance (0.5 mm) for an Al test sample with no excitation (AluDC1 and AluDC2904-b curves) and 10V DC excitation.

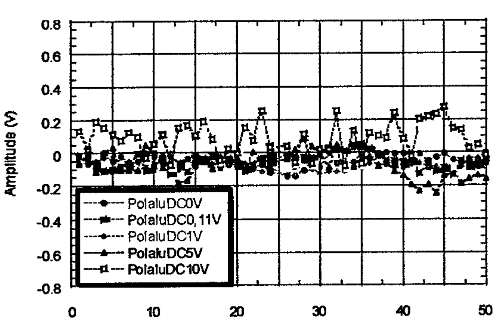



Figure 8 : Comparison of the signals for the same lift-off distance (0.5 mm) for Al charged (30%) polymer test sample for different DC excitation (0 to 10V).

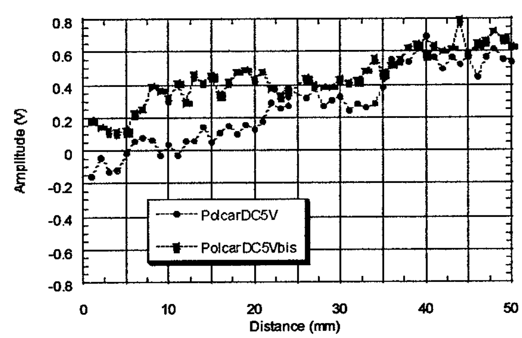



Figure 9 : Comparison of the signals for the same lift-off distance (0.5 mm) for Carbon charged (5%) polymer test sample for a 5V DC excitation signal.

## IV.3. AC measurements

An AC magnetic excitation signal has been applied using the Helmholtz coils connected to a function generator and a lock-in amplifier has been used to achieve a synchronous detection of the eddy current induced in different materials. The impedance of the coils has been characterized previously : $Z = R + j L \omega$ with $R=6.4\ \Omega$ and $L=33.16$ mH. The frequency of the excitation signal is fixed at 180 Hz at which there is no noise peak in the environment

First, the Plexiglas support has been measured alone with no sample on it at 2mm from the SQUID and Figure 10 shows that no signal is obtained for an AC excitation signal of 1 volt peak to peak (pp) corresponding to a magnetic field amplitude of 0.12 G pp or 43 mG RMS. Then an Al sample positioned on the Plexiglas support and at 2mm du SQUID for the same AC excitation showed a reproducible signal (Figure 11). In order to study the influence of the lift-off distance, the variation of the signal obtained with the Al sample for the same AC excitation signal is recorded from 0.5 to 10.2 mm and this has been compiled in figure 12.

The comparisons of the signals obtained for the same lift-off distance (0.5 mm) and the same strong AC excitation signal (3.5 volt pp corresponding to a magnetic excitation of 0.425 G pp or 0.15 G RMS) for Al and two other test samples (polymers with 30% Al particles and 5% Carbon nanoparticle) are given by the figure 13. Despite the presence of the conducting particles, the polymer samples seem to have no Eddy current effect.

To show the crack detection of our system, four thick Al sheets (110 X 100 X 5 mm$^3$) have been elaborated with three of them having cracks in the middle of their upper surface with the same depth (1mm) and different width (0.5 to 2 mm). The measurements have been done with a lift-off distance of 0.5 mm and a magnetic excitation of 0.15 G RMS. Figures 14.A and 14.B show that even the smallest crack can be detected when it is on the lower face of the Al thick sheet.

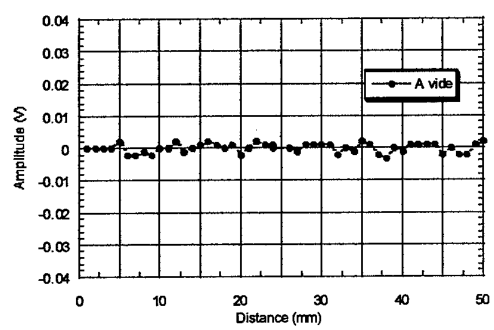



Figure 10 : No signal obtained for the Plexiglas support alone at 2mm du SQUID for a AC excitation signal.

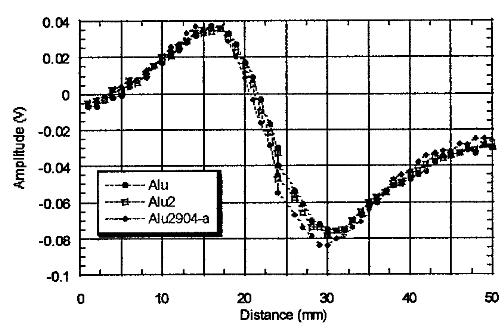



Figure 11 : Reproducibility of the signal obtained for Al test sample at 2mm du SQUID for a AC excitation signal.

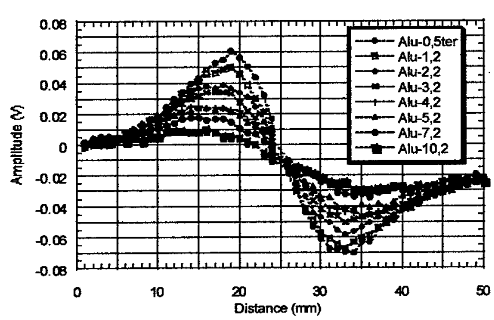



Figure 12 : Variation of the signal with the lift-off distance (0.5 to 10.2 mm) for Al test sample for the same AC excitation signal.

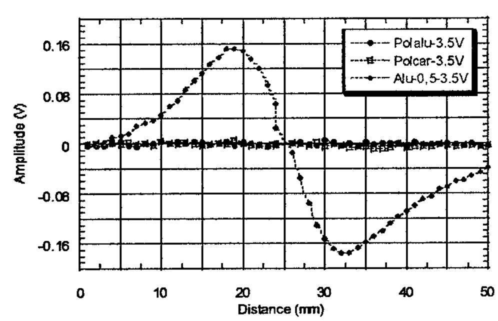
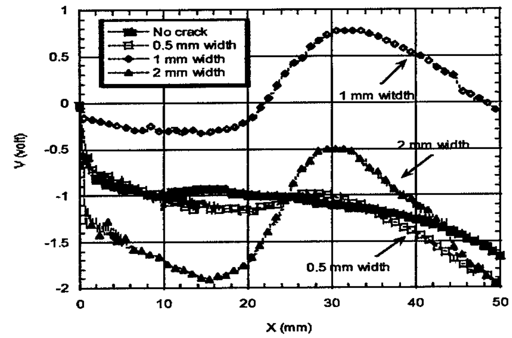



Figure 13 : Comparison of the signals for the same lift-off distance (0.5 mm) and the same strong AC excitation signal for Al and two other test samples (polymers with 30% Aluminum and 5% Carbon particles).



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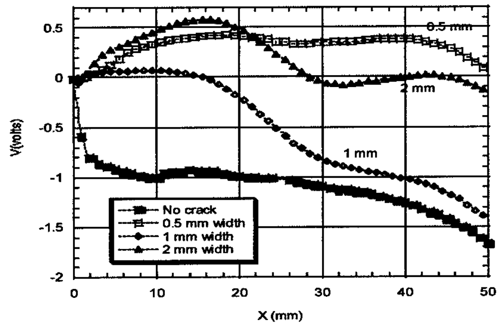

B

Figure 14 : Variation of the signal obtained for different crack width (0.5 to 2 mm) with the same depth (1mm) on the upper (A) and lower (B) faces of a thick Al sheet (110 X 100 X 5 mm3). The lift-off distance is 0.5 mm, and the AC magnetic excitation has an RMS value of 0.15 G.

## V. Conclusions and perspectives

The SQUID NDE experiment has been set up successfully in our laboratory. Despite the fact that one of the four SQUIDs (Z2 one) was damaged only after five months and this created several months of misfunctionning of the whole system, we could achieve NDE tests after the replacement of the damaged SQUID by the JSQ company.

The LABVIEW programs have been optimized to do precise scanning stage movement control and data acquisition, and so they allow an accurate surface inspection of different materials.

The noise figures of the SQUIDs show the utility to acquire a shielded 3-layer mu-metal cylinder in order to reduce the DC and low frequency noise during the NDE experiments.

Preliminary results show that cracks as small as 0.5 mm width and 1mm depth can be detected. Other tests are in progress to optimize the experimental conditions and to show the possibility of detection of smaller-sized flaws. Nevertheless, as we would like to increase the resolution of the system, we are considering reducing the total lift-off distance of our SQUID NDE system by changing the dewar configuration provided by JSQ company.

Concerning the materials to study, magnetic-particles-filled polymers have been considered, and an intensive investigation of the literature has revealed no work in this field, but did uncover in magnetic particle inspection (on the surface of the flaws) decoration method [5]. Moreover, we would like to engage for the next fall a Ph.D. student to study, by our SQUID NDE system, the mechanism of crack formation and propagation in composites-based organic matrix materials, as well as in aluminum and carbon-composite material used in aircraft structures.

Additionally, this project has created interest at our university for SQUID MCG applications, and contacts have been made with CMI (Cardiomag Imaging), in order to investigate the possibility of acquisition of an MCG system by our university in a collaboration between the medical and electrical engineering departments.

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